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LIPID RESIDUES FROM LOW-FIRED POTTERY*

E. A. REBER,[1]† M. T. KERR,[2] H. L. WHELTON[3] and R. P. EVERSHED[3] Q1

[1]*Anthropology Department, UNC Wilmington, 601 S. College Road, Wilmington, NC 28403, USA*

[2]*Department of Geography, The University of Tennessee, 304 Burchfield Geography Building, Knoxville, TN 37996, USA*

[3]*Organic Geochemistry Unit, School of Chemistry, University of Bristol, Bristol BS8 1TS, UK*

*This paper assesses the fate of lipids associated with low-temperature and pit-fired pottery to determine to what degree organic compounds persist or are removed during short-firing episodes below 800 °C. Three different types of clay were fired using contrasting techniques including at 400 °C for 4 h in a muffle furnace, and pit-firing in which pottery was fired to higher temperatures but for shorter periods of time. Total lipid extracts obtained by solvent extraction of test sherds were screened using gas chromatography-flame ionization detection (GC-FID) to determine the lipid concentrations and gas chromatography–mass spectrometry (GC–MS) to identify the organic compounds present. The results showed that firing of clay removes all naturally occurring alkyl lipids; however, during pit-firing, diterpenoid lipids were introduced into the clay as a condensate from pine (*Pinus *spp.) wood used as fuel. These results confirm that alkyl lipids, e.g., fatty acids, can be reliably associated with the use of vessels, although caution is required when interpreting the origins of lipids that might derive from fuel used in firing.*

KEYWORDS: POTTERY, LIPID RESIDUE ANALYSIS, GAS CHROMATOGRAPHY–MASS SPECTROMETRY, QUANTIFICATION, ALKYL LIPIDS, DITERPENOIDS

INTRODUCTION

Lipid residue analysis of absorbed residues from archaeological pottery is one of the few analytical methods that can directly link a pot to its original contents, and hence to wider aspects of food procurement and processing. Lipid residues are absorbed into the matrix of a pot during its use. Once absorbed, lipid residues are well protected from chemical decay and microbial attack (Heron *et al*. 1991; Dudd *et al*. 1999). These components can originate from a variety of sources, including terrestrial animal products (Evershed *et al*. 1991a; Copley *et al*. 2003), leafy vegetables (Evershed *et al*. 1991b, 1994), aquatic commodities (Hansel *et al*. 2004; Evershed *et al*. 2008; Cramp *et al*. 2014b) and plant resins (Charters *et al*. 1993a; Stern *et al*. 2003; Reber and Hart 2008; Reber and Kerr 2012; Baeten *et al*. 2014). The high proportion of saturated hydrocarbon moieties present in lipids reduces their structural modification and degradation over time owing to their inherent hydrophobicity and entrapment within a ceramic matrix, thereby making them excellent candidates as biomarkers (Evershed 1993, 2008a). Archaeological lipid residues are interpretable because most lipids naturally occurring in clays are thought to be removed during firing, leaving a 'blank slate' for absorption of culturally produced lipids during pot use (de Atley 1980; Stott *et al*. 2003; Berstan *et al*. 2008; Evershed 2008a).

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†Corresponding author: email rebere@uncw.edu

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Considerable research has taken place on these topics and the theory behind residue analysis is well understood (Charters *et al*. 1993b, 1997; Evershed *et al*. 1991a, 1994, 1995, 1997; Dudd *et al*. 1998; Hansel *et al*. 2004; Mukherjee *et al*. 2005; Hansel and Evershed 2009; Correa-Ascencio and Evershed 2014). The interpretation of absorbed residues, however, is recognized as being complicated by several factors (Evershed 2008b; Roffet-Salque *et al*. 2017). First, the variety of potential sources for anthropogenic residues described above means that residues may not be assumed to derive simply from pot contents, as studies have shown post-firing applications of resin for use as a sealant (Regert *et al*. 2003; Reber and Hart 2008; Stern *et al*. 2008; Stacey *et al*. 2010; Correa-Ascencio *et al*. 2014), as a glue (Charters *et al*. 1993a; Urem-Kotsou *et al*. 2004; Connan *et al*. 2008) and for decoration (Connan *et al*. 2004). Second, since a pot is generally used to process a variety of resources over its use-lifetime (Evershed 2008b), and many of these resources have overlapping suites of lipids, the result is that archaeological residues usually contain at least some lipids deriving from multiple resources (Frith *et al*. 2004; Reber *et al*. 2004; Cramp *et al*. 2011, 2014a; Dunne *et al*. 2012). Third, once a residue-containing ceramic is buried, a variety of further diagenetic reactions may occur that cause changes to lipid components of the absorbed residue including oxidation, hydrolysis and microbial decomposition (Evershed and Charters 1995; Dudd *et al*. 1998; Malainey *et al*. 1999). Owing to the complexity of archaeological residue interpretation, it is essential that analysts first determine whether extracted lipids are interpretable and represent cultural signals, or if instead the lipids are a result of deposition or post-excavation contamination. The impact of post-excavation contamination can be reduced through mechanical removal of the sherd surface before analysis (Condamin *et al*. 1976; Heron *et al*. 1991) and contamination from the migration of soil lipids into the pottery vessels during burial has been shown to be distinctly different from lipids that arise from vessel use (Heron *et al*. 1991).

The present paper presents the results of investigations aimed at determining potential sources of surviving lipids in low-fired pottery. We fired three different types of clay using a variety of techniques. These included a firing at 400 °C for 4 h in a muffle furnace, and pit-firing in which pottery samples were fired to higher temperatures but for generally shorter periods of time. The obtained total lipid extracts (TLEs) from the analyses of the sherds were screened using gas chromatography-flame ionization detection (GC-FID) in order to determine the lipid concentrations and with gas chromatography–mass spectrometry (GC–MS) to identify the organic compounds present.

MATERIALS AND METHODS

Three different clays were used for experimental firing. The first was commercial low-firing red earthenware (Standard Ceramic Supply Company 103 Red Clay) purchased from Fat Cat Pottery (Wilmington, NC, USA). The second was a dark, organic-rich clay found washed up on the Atlantic coast at Fort Fisher, NC, USA, after a storm (sea clay). In coastal regions of the eastern seaboard of North America, this is one of the most reliable and easily obtained sources of clay for potters, as much of the terrestrial clay is deeply buried under layers of sand. The third was a terrestrial clay excavated from under layers of sand in Wilmington, NC, USA. All clays were fired as tiles, with no additional temper added. All tiles were approximately 9.0 × 12.0 × 0.5 cm in measurement, and were dried thoroughly at room temperature before firing.

The clay tiles were fired using two methods: in a muffle furnace at 400 °C for 4 h (firing 1) and in several firing patterns in earthen pits excavated in the ground (firings 2–4). The pit-firing temperatures were measured with a Bailey Type K Chromel-Alumel Analog pyrometer (accuracy

± 2%), whose thermocouple was placed next to the red earthenware tile. The ceramic tiles were fired unwrapped, balanced on firebricks in the base of the pit, with all tiles located equal distances from the centre of the pit, and equal heights from the base of the pit. All pit-firings used a mixture of pine (*Pinus* spp.) and hardwoods, primarily oak (*Quercus* spp.). Pit-firing continued until the fires were well established and the pyrometer indicated that the temperature was consistently above 400 °C. Differences in weather, wood and wind conditions resulted in firings at various temperatures and for varying lengths of time before a cover was placed on the pit to produce a reducing atmosphere while the pottery cooled (Fig. 1). In general, the pit-firings were distinguished by rapid spikes in temperature, followed by an equally rapid fall in temperature after the changeover to reducing conditions. All firing temperatures were sufficient to produce fired clay tiles and pottery (Cardew 1969). **F1**

Water is largely driven off from the clay early in the firing process. Surface-absorbed water is removed by 200–300 °C and the removal of lattice water (dehydroxylation) can vary between 300 and 800 °C. Similarly, the oxidation of carbon begins at around 200 °C resulting in the blackening of the clay as carbon moves to the surface. At temperatures above 600 °C (usually in excess of 750 °C) all carbon is eliminated from the clay (Rice 1987). All clay types after firings 1 and 3 had a slightly darkened appearance (compared with the unfired clay). After firings 2 and 4, all clay types were visibly blackened and smelled strongly of wood smoke. Unfired clays were wrapped in aluminium foil to prevent dust contamination and dried at room temperature before sampling.

Samples of unfired clays and clay tiles from all firing types were submitted to lipid analysis following Correa-Ascencio and Evershed (2014). Briefly, a 2 g portion of the exterior surface of each sample was cleaned using a modelling drill to remove modern surface contaminants. Cleaned samples were subsequently crushed and ground to a fine powder using a pestle and mortar. A known amount of internal standard (*n*-tetratriacontane, 20 µl, 1.0 mg ml^{-1} solution) was added to the powder. Lipids were then extracted using 5 ml of 4% sulfuric acid/methanol

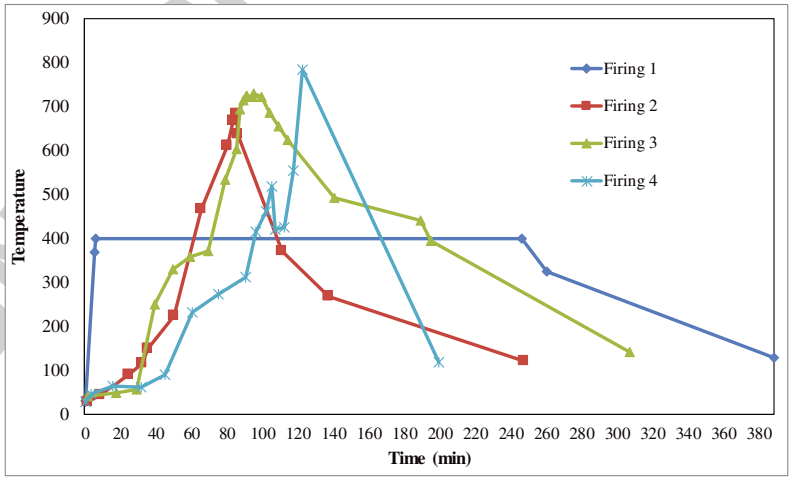

Figure 1 *Temperatures and durations for all firings described in the text. Firing 1 was done in a muffle furnace, while firings 2–4 were pit-fired using a mixture of pine (*Pinus *spp.) and hardwood, primarily oak (*Quercus *spp.)*. [Colour figure can be viewed at wileyonlinelibrary.com]

solution ($\delta^{13}C$ measured), which also hydrolyzed and transmethylated extracted acyl lipids to fatty acid methyl esters (FAMEs). Before analysis by GC-FID and GC–MS, an aliquot of the extract was derivatized with *N*,*O-bis*(trimethylsilyl)trifluoroacetamide (BSTFA) containing 1% trimethylchlorosilane (TMCS; Sigma Aldrich) to derivatize neutral lipids in the extract to trimethylsilyl (TMS) esters. The derivatized TLEs were screened using GC-FID in order to determine the lipid concentrations. The extracts containing organic compounds were further analyzed using GC–MS for identification.

Analyses of TLEs prepared as above were performed using an Agilent 7820A GC. The FID used to monitor the column effluent was set to 300 °C. Samples were introduced to the system via on-column injection (1.0 µl). The analytical column was a 50 m × 0.32 mm fused silica capillary column coated with an HP-1 non-polar stationary phase (100% dimethylpolysiloxane, 0.17 µm; Agilent J&W Scientific). The GC temperature programme was held at 50 °C for 2 min, followed by a gradient increase to 300 °C at 10 °C min^{-1}, and the oven was then run isothermally for 15 min. Helium was used as the carrier gas set to a constant flow of 2.0 ml min^{-1}. Data were acquired using HP Chemstation software (Rev. C.01.07; Agilent Technologies) and eluted peaks were identified by comparison of retention times with those of an external standard. Quantification was based on the known amount of internal standard introduced during sample preparation.

GC–MS analyses of TLE aliquots were performed using a ThermoScientific Trace 1300 GC coupled to an ISQ single quadrupole MS. Samples were introduced via a PTV injector set Q3 Q4 to splitless mode onto a 50 m × 0.32 mm fused silica capillary column coated with an Rtx-1 stationary phase (100% dimethylpolysiloxane, 0.17 µm; Restek) for non-polar analyses. The GC temperature programme was set to hold at 50 °C for 2 min, followed by a gradient increase to 300 °C at 15 °C min^{-1}. Once at 300 °C, the oven was run isothermally for 15 min. He was used as the carrier gas, set to a constant flow of 2 ml min^{-1}. The MS was operated in electron ionization (EI) mode operating at 70 eV, with a GC transfer line temperature of 300 °C and a source temperature of 300 °C. The emission current was set to 150 µA and the MS was set to acquire in the range of *m/z* 50–650 at 2 scans s^{-1} in full-scan mode. Data acquisition and processing were carried out using XCalibur software (v. 3.0). Compounds were identified by comparison with the National Institute of Standards and Technology (NIST) mass spectra library (v. 2.0) or with Q5 reference to external sources such as The Lipid Library (www.lipidlibrary.aocs.org)

RESULTS

Analysis of the unfired pottery revealed that the sea clay and terrestrial clay contained 193 µg lipid per gram of sample (µg g^{-1}) and 60 µg g^{-1} respectively (Fig. 2). The presence of lipids in F2 the unfired clay is likely a reflection of the source of the clay. The sea clay contained large quantities of cyclic octa-atomic sulfur from the anaerobic respiration of sulfur-reducing bacteria, indicating the clay derived from a waterlogged/underwater environment (Schauder and Kroger 1993; Fors *et al*. 2014). In addition, both the sea clay and terrestrial clay contained lipids derived from plant origin (Eglinton and Hamilton 1967; Tulloch 1976), including long-chain fatty acids (LCFAs) of carbon chain lengths C_{13}–C_{34} in the sea clay and C_{16}–C_{29} in the terrestrial clay. Additionally, *n*-alcohols of carbon chain length C_{18}–C_{26} were detected in the sea clay, and *n*-alkanes of carbon chain lengths C_{21}–C_{31} were detected only in the terrestrial clay. Both the sea and terrestrial clays contained unidentified terpenoids. Contrastingly, the red earth clay contained trace levels of lipids (< 5 µg g^{-1}).

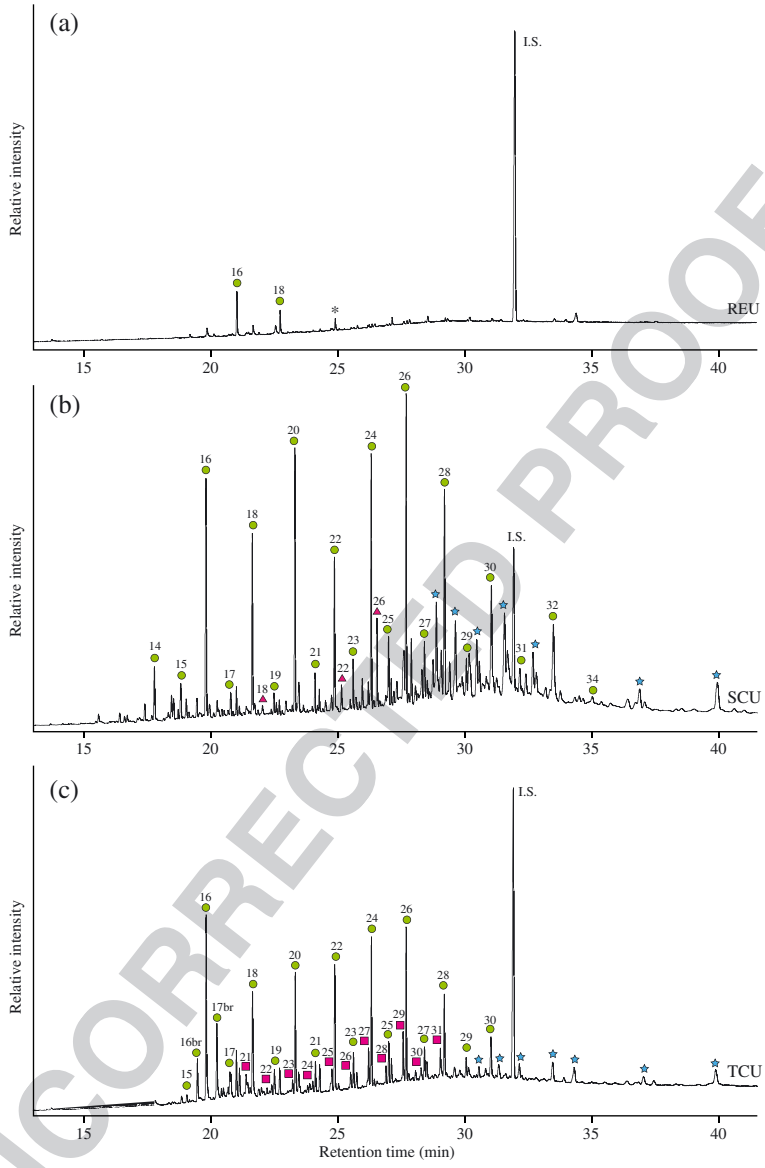

Figure 2 *Partial gas chromatography profile of the acid-extracted total lipid extracts of the unfired clays; circles,* n-*alkanoic acids (fatty acids); squares,* n-*alkanes; triangles,* n-*alcohols; stars, terpenoids; IS, added internal standard (C_{34}* n-*alkane); *plasticizer contamination; RE, red earthenware; SC, sea clay; and TC, terrestrial clay.* [Colour figure can be viewed at wileyonlinelibrary.com]

After firing 1, none of the clay types contained significant detectible lipids ($< 5\,\mu g\,g^{-1}$; Table 1 and Fig. 3). Firing 1 reached a constant 400 °C in a muffle furnace, and lasted for 4 h before gradual cooling (Fig. 1).

After firing 2, increased lipid concentrations were observed in all clay types. Lipid concentrations detected were $95\,\mu g\,g^{-1}$ in the red earth clay, $362\,\mu g\,g^{-1}$ in the terrestrial clay and $1128\,\mu g\,g^{-1}$ in the sea clay (Table 1 and Fig. 4). Most of the lipids identified derived from

Table 1 *Summary of the analyses of fired clay samples*

Clay	*Sample*	*Lipid concentration* ($\mu g\,g^{-1}$)	*Firing method*
Red earthenware	REU	2.6	–
	REF1	1.0	Muffle furnace
	REF2	94.5	Pine and hardwood earthen pit
	REF3	1.8	Pine and hardwood earthen pit
	REF4	96.2	Pine and hardwood earthen pit
Sea clay	SCU	193.3	–
	SCF1	2.0	Muffle furnace
	SCF2	1128.3	Pine and hardwood earthen pit
	SCF3	2.3	Pine and hardwood earthen pit
	SCF4	502.6	Pine and hardwood earthen pit
Terrestrial clay	TCU	60.7	–
	TCF1	1.3	Muffle furnace
	TCF2	361.9	Pine and hardwood earthen pit
	TCF3	1.2	Pine and hardwood earthen pit
	TCF4	7.9	Pine and hardwood earthen pit

the firing of the pottery in wood-fired earthen pits. The most abundant compounds were products of the pyrolysis of wood and the degradation of diterpenoid acids. Comparisons with reference mass spectra identified the major components (Fig. 5) as retene, 18-norabieta-8,11,13-triene, 19-norabieta-8,11,13-triene, methyl dehydroabietate, dehydroabietic acid (TMS ester), methyl-6-dehydroabietate and methyl 7-oxodehydroabietic acid (Evershed *et al*. 1985; Robinson *et al*. 1987; Simoneit *et al*. 1993; Mills and White 1994; Berg *et al*. 2000; Colombini and Modugno 2009). No LCFAs, *n*-alkanes, *n*-alcohols or triterpenoids were detected. A large amount of cyclic octa-atomic sulfur was observed in the sea clay pottery. F5

Only trace amounts of lipids were detected in all clay types after firing 3 ($< 5\,\mu g\,g^{-1}$), which briefly reached 730 °C, with a longer, hotter cooling period than the other pit-firing episodes (Fig. 1). Lipid profiles were comparable with those after firing 1 (Table 1 and Fig. 3).

After firing 4, increases in lipid concentrations were observed in the red earth and sea clays. Lipid concentrations of $96\,\mu g\,g^{-1}$ were detected in the red earth clay and of $502\,\mu g\,g^{-1}$ in the sea clay. Most of the lipids identified again derived from firing the pottery in wood-fired earthen pits. The most abundant compounds were again products of the pyrolysis of wood and the degradation of diterpenoid acids. These included: retene, 18-norabieta-8,11,13-triene, 19-norabieta-8,11,13-triene, methyl dehydroabietate, dehydroabietic acid (TMS ester), methyl 6-dehydroabietate and methyl 7-oxodehydroabietic acid. A significant amount of cyclic octa-atomic sulfur was again observed in the sea clay pottery. No LCFAs, *n*-alkanes, *n*-alcohols or triterpenoids were detected in any pottery types. Some variation was observed in how the different clays reacted to the same firing conditions, as only trace amounts of lipids were detected in the pottery made with terrestrial clay ($7\,\mu g\,g^{-1}$). The lipid profiles were analogous to those observed after firing 2 (Fig. 4).

DISCUSSION

Firing the pottery tiles appeared to remove lipids originally present in the unfired clays, as only trace concentrations of lipids were observed in the pottery after firings 1 and 3. This suggests that

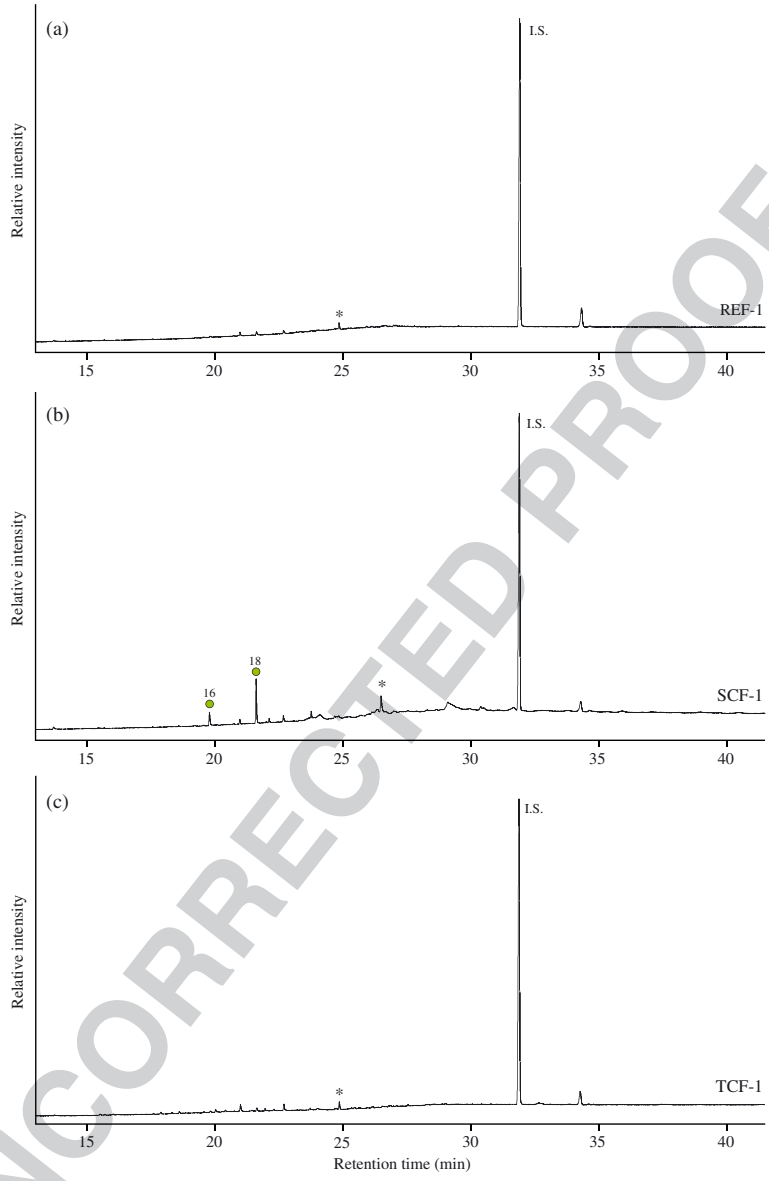

Figure 3 *Partial gas chromatography profile of the acid-extracted total lipid extracts for firing 1 of all clays; circles,* n-*alkanoic acids (fatty acids); IS, added internal standard (C_{34}* n-*alkane); *plasticizer contamination; RE, red earthenware; SC, sea clay; and TC, terrestrial clay*. [Colour figure can be viewed at wileyonlinelibrary.com]

in the case of archaeological pottery, any lipids recovered from the analyses above the threshold of 5 $\mu g\, g^{-1}$ would not derive from the original clay used to manufacture the pottery.

Lipids were recovered following firings 2 and 4, with GC–MS analyses of the lipids indicating that the compounds were not residues or thermal alteration products of naturally occurring lipids in the parent clays. Rather, they were produced from the oxidation and thermal degradation of

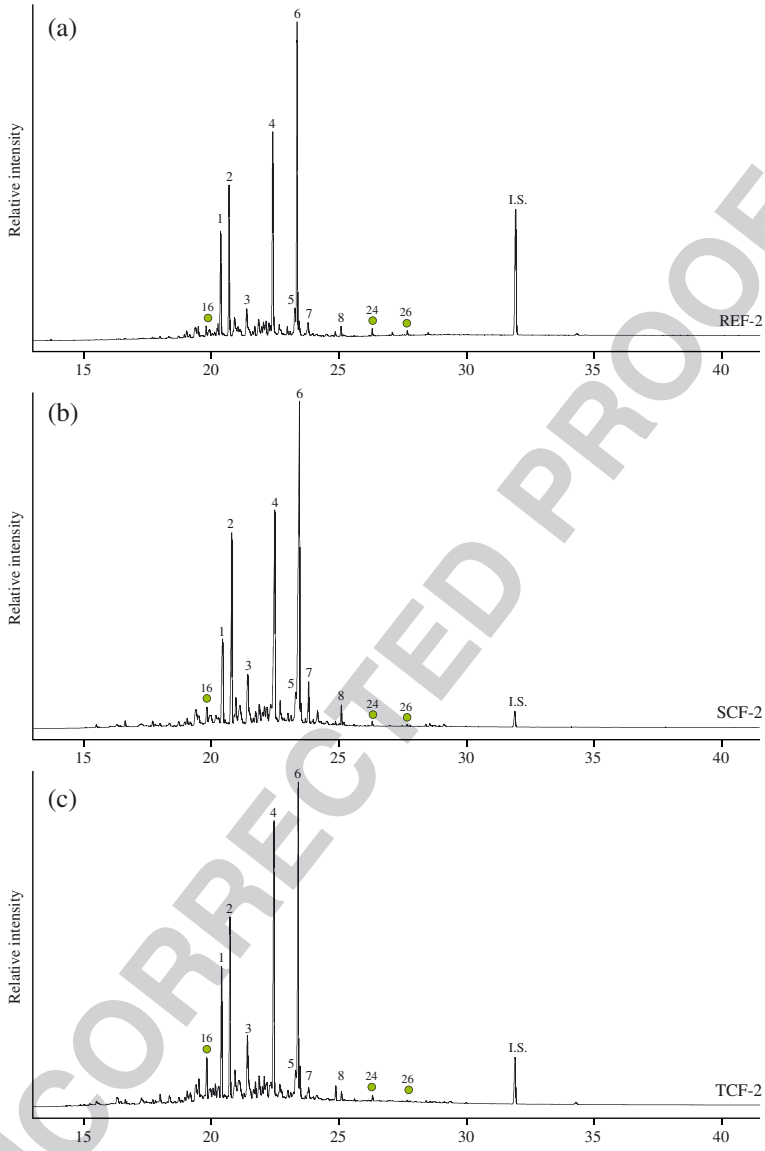

Figure 4 *Partial gas chromatography profile of the acid-extracted total lipid extracts for the firing 2; circles,* n-*alkanoic acids (fatty acids); 1: 18-norabieta-8,11,13-triene; 2: 19-norabieta-8,11,13-triene; 3: 1,2,3,4-tetrahydroretene; 4: retene; 5: methyl-6-dehydroabietate; 6: methyl dehydroabietate; 7: dehydroabietic acid (TMS); 8: methyl 7-oxodehydroabietic acid; IS, added internal standard (*C_{34} n-*alkane); RE, red earthenware; SC, sea clay; and TC, terrestrial clay*. [Colour figure can be viewed at wileyonlinelibrary.com]

pine wood resin (Fig. S1). The most abundant compounds identified included 18-norabieta-8,11,13-triene, 19-norabieta-8,11,13-triene, retene and methyl dehydroabietate (Evershed *et al*. 1985; Robinson *et al*. 1987; Simoneit *et al*. 1993; Mills and White 1994; Berg *et al*. 2000; Colombini and Modugno 2009). These compounds are all products of the thermal dehydrogenation of abietic acid naturally present in the wood, indicative of the heating of pine wood to high

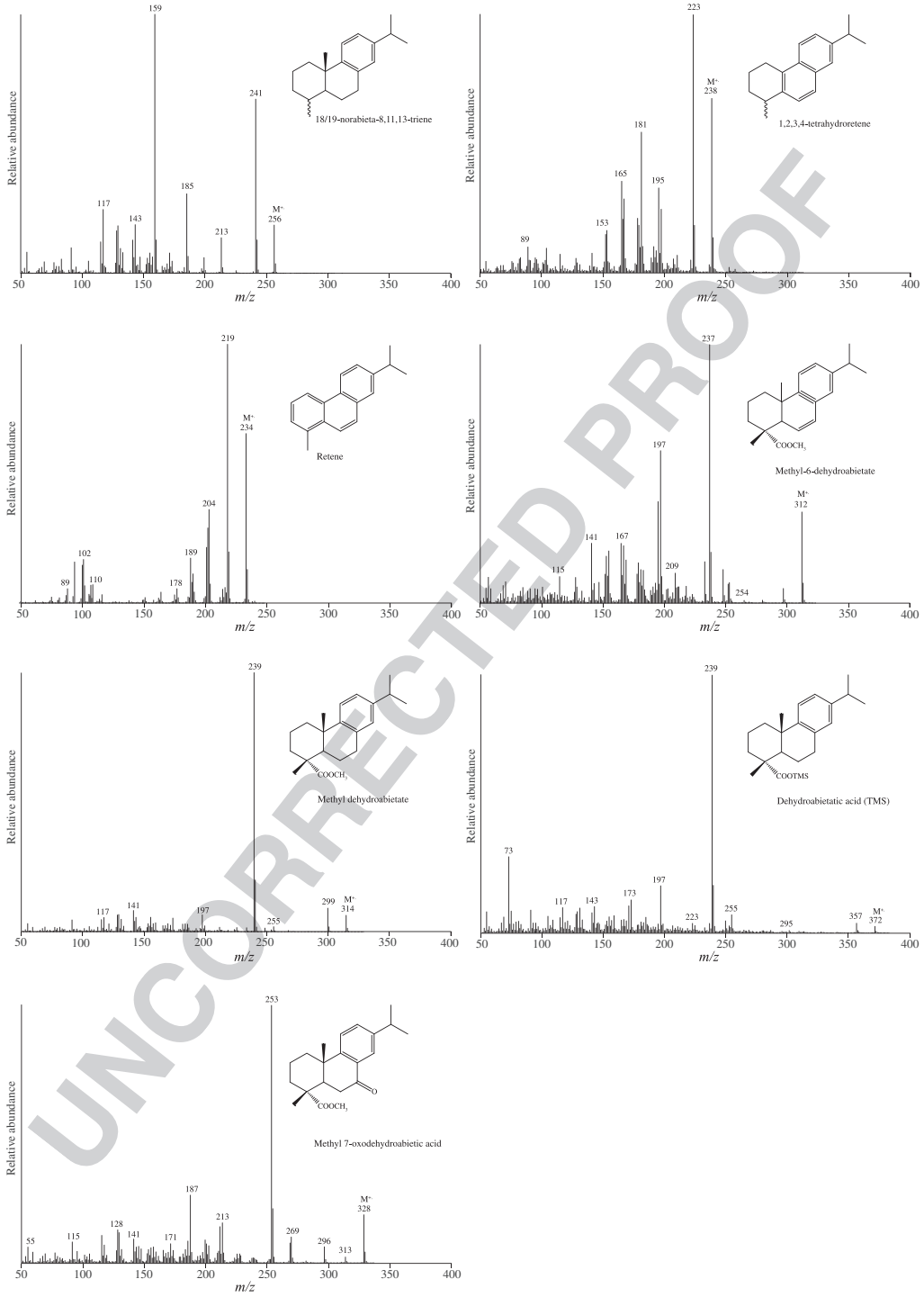

Figure 5 *Mass spectra of the major diterpenoids identified.*

temperatures (Evershed *et al*. 1985; Colombini and Modugno 2009; Stacey *et al*. 2010). This heating occurred in an oxygenated environment, as evidenced by the presence of methyl 7-oxodehydroabietic acid (Colombini and Modugno 2009). The presence of these compounds can, therefore, be unambiguously attributed to smoke condensate from firing using pine.

Firing temperature, firing time and the organic composition of clay have been shown to be important variables in the removal of naturally occurring alkyl lipids from clay (Johnson *et al*. 1988). The results obtained herein confirm that firing removed all naturally occurring lipids from the clay in this study; however, lipids from the pyrolysis of wood were introduced into the clays after two of the three pit-firings. All wood firings reached temperatures greater than 400 °C (the temperature at which organic carbon is driven off from the clay) for periods of 45, 120 and 80 for firings 2, 3 and 4 respectively (Fig. 1). In addition, all wood firings reached temperatures greater than 700 °C, which should have effectively eliminated all carbonaceous material in the clay (Rice 1987). The observed differences between wood fired clays (firings 2–4) can be explained by the differences in firing temperature and times. Lipids derived from wood pyrolysis were introduced to all clay types after firings 2 and 4, but not after firing 3. While firing 4 reached higher temperatures than firing 3, firing 3 maintained a temperature above 700 °C for a longer period. Similarly, the cooling period of firing 3 was longer and hotter than both firings 2 and 4. This longer firing time at a higher temperature likely ensured that all lipids, either naturally occurring or resulting from pyrolysis during firing, were removed from the clay. An additional possibility is that the slow cooling period in firing 3 was due to a more abundant presence of oxygen within the firing pit during the cooling down period, resulting in a more oxidizing atmosphere than in firings 2 and 4. Normally, closing the pit to produce a reducing atmosphere results in a very rapid cooling down (Fig. 1), which was not the case in firing 3. Additionally, pottery from firing 3 was redder and smelled less obviously of smoke upon removal from the pit, which is a potential further indicator of the accidental addition of oxygen to the firing pit during the cooling down period. Either the longer period at higher temperatures or a combination of higher temperatures with more oxygen likely resulted in the further oxidation of pyrolysis condensates into volatile compounds that did not deposit on the pottery.

Considerable variation was observed in pyrolytically produced lipids within tiles from the same firing episode. For example, as described above, firing 2 introduced lipid concentrations of 95 $\mu g\, g^{-1}$ to the red earth clay, 362 $\mu g\, g^{-1}$ to the terrestrial clay and 1128 $\mu g\, g^{-1}$ to the sea clay, while firing 4 introduced 96 $\mu g\, g^{-1}$ to the red earth clay, only 7.9 $\mu g\, g^{-1}$ to the terrestrial clay and 502 $\mu g\, g^{-1}$ to the sea clay. This intra-firing variation is, therefore, not due solely to the physicochemical structures of the differing clays. Rather, these differences were likely due to variations within each pit-firing. The pit-firings were constructed to provide as much similarity as possible in firing condition around the three tiles with respect to wood placement and air circulation. It was impossible to produce perfect similarity, however, given variations in wood shape and size and unexpected breezes around the firing site. All firings included hot and cold spots, not all of which could be measured by the thermocouple of the pyrometer. The relatively small concentration of pyrolytic lipid added to the terrestrial clay in firing 4, for example, was likely due to a hot spot that resulted when a flaming log unexpectedly collapsed on the terrestrial clay tile, but not the red earth tile, where the thermocouple was placed. The short period of extreme heat not captured by the pyrometer would have oxidized and volatilized condensates from much of the affected tile. Considerable variation in temperature within open-pit-firings has been previously noted in the literature (Gosselain 1992). Other sources of variation were also present within each firing. The different amounts of pyrolytically added lipids in firing 2, for example, may derive from air currents within the fire, which would have affected smoke condensation at different

spots, or from the varying proximity of the tiles to particularly resinous pieces of pine wood. Such variation is normal within pit-firings, and can be expected within archaeologically pit-fired pots.

In this study, pyrolysis products were derived overwhelmingly from combustion of *Pinus* spp., even though both pine and hardwood were used in all pit-firings. This is probably because most of the hardwood used in the firings in this project was from oak (*Quercus* sp.), which is not a resinous wood (Langenheim 2003, 74–6). Pine, on the other hand, is a well-known source of abundant diterpenoid resins (Mills and White 1994; Langenheim 2003, 54–7). Pottery fired with resinous wood, such as from *Pinus* spp. would therefore be particularly prone to potential misinterpretation of pyrolytic byproducts from firing. For example, biomarkers of pine resin in a residue could derive from deliberate application as part of a post-firing treatment (Reber and Hart 2008), from pine pitch processing (Hjulstrom *et al*. 2006; Reber and Hart 2008), or from by-products of fuel used in firing.

Since this study used only pine and mixed hardwoods (predominantly oak) in firing, it did not evaluate the potential input from other resinous or organic firing fuels, such as birch, resinous shrubs or dung. Pyrolytic lipids from fuel sources in low-temperature pottery likely vary based on the individual type or mixture of fuel used in firing. Future studies will need to determine the amount and kind of pyrolytic products from a wider variety of resinous or organic fuels.

CONCLUSIONS

Lipid residue analyses in this study demonstrated that unfired clays contain significant concentrations of background lipids; however, these lipids were removed during firing of the clays. Firing using a muffle furnace was most effective for removing the background lipids. The temperature of the firings did not appear to affect the removal of background lipids, with removal being complete at all temperatures. However, new lipids were introduced into the pottery during wood firings. The presence of diterpenoid biomarkers in the fired pottery tiles that were not present in the clays before firing clearly indicates that the pyrolysis of wood resulted in the transfer of smoke condensates into pottery during firing. Diterpenoid biomarkers in pottery residues can, therefore, derive from the firing process, as well as from deliberate use of pine resins. It is important to conclude, therefore, that biomarkers for wood or fuel pyrolysis should be interpreted cautiously, and not immediately used to indicate resource processing or use within the pot. Structural alterations that occur as a result of processing, such as intense heating, can provide insights into how the biomarkers were produced (Evershed *et al*. 1985; Evershed 2008b). In addition, future studies are needed to determine what other pyrolytic products of fuel may condense on pottery during firing, and in what quantities.

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SUPPORTING INFORMATION

Additional Supporting Information may be found online in the supporting information tab for this article.

Figure S1. Oxidation and thermal degradation products of pine wood. Sources: Adapted from Pastorova (1997) and Colombini (2009). Q2